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PATENT

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In re application of: Miller et al.

MAR 25 2004

Application No.: 10/680,852

Group No.:

Filed: 10/7/2003

For: Homogeneous Microemulsion Comprising Polyethylene Glycol

Commissioner for Patent

P.O. Box 1450

Alexandria, VA 22313-1450

INFORMATION DISCLOSURE STATEMENT

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Applicant: Miller et al.
Serial No. 10/680,852
Page 2

Dear Sir:

In accordance with the duty of disclosure provisions of 37 CFR §1.56, there is hereby provided certain information which the Examiner may consider material to the examination of the subject U.S. patent application. It is requested that the Examiner make this information of record if it is deemed material to the examination of the subject application. No admission is made that the information cited in this Statement is, or is considered to be, material to patentability nor a representation that a search has been made (other than search report(s) from a counterpart foreign application or a PCT International Search Report, if submitted herewith).

1. ☒ Enclosures accompanying this Information Disclosure Statement are:
 - 1a. ☒ Form PTO-1449.
 - 1b. ☒ Copies of Information Disclosure Statement citations.
 - 1c. ☒ EP Search Report number EP 03 02 2145 dated November 11, 2003.
 - 1d. ☒ Copies of Co-pending U.S. Applications.
2. ☐ This Information Disclosure Statement is filed under 37 CFR §1.97(b) with the filing of the U.S. patent application. Accordingly, no fee or §1.97(e) Statement is required.
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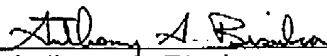
Applicant: Miller et al.

Serial No. 10/680,852

Page 3

7. ☒ The English language documents identified by reference in the TRANSLATION Section of the foreign patents list of the attached 1449 Form are believed to be a concise statement, or the equivalent or substantial equivalent to the foreign language documents cited in the attached 1449 Form.
8. ☒ In addition to the documents cited, the Office's attention is directed to the following co-pending U.S. Application Serial Number(s), copies of which are attached for the convenience of the Office:
- a. U.S. serial no. 10/338,452, filed 1/8/2003, Miller et al.

Respectfully submitted,



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Page 1 of 1

FORM PTO-1449 U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE INFORMATION DISCLOSURE STATEMENT BY APPLICANT (37 CFR 1.98(b))	ATTY. DOCKET NO. 2002DE434	SERIAL NO. 10/680,852
	APPLICANT: Miller et al.	
	FILING DATE: 10/7/03	GROUP:

U.S. PATENT DOCUMENTS

Examiner Initial		Patent Number	Issue Date	Patentee	Class	Subclass	Filing Date, If Appropriate
	AA	6,562,356	5/13/03	Verite et al.	424	401	
	AB						
	AC						
	AD						

FOREIGN PATENT OR PUBLISHED FOREIGN APPLICATION

		DOCUMENT NUMBER	PUB. DATE	COUNTRY		TRANSLATION
	BA	DE 38 25 539	1/26/89	Germany		See CA below.
	BB	DE 196 12 085	10/2/97	Germany		See CB below.
	BC	EP 1 327 439	7/16/03	Europe		See IDS Paragraph 8.a.
	BD					
	BE					

OTHER DOCUMENTS (Including Author, Title, Date, Place of Publication**)

	CA	English abstract of DE 38 25 539.
	CB	English abstract of DE 196 12 085.
	CC	
	CD	
EXAMINER		DATE CONSIDERED
EXAMINER: Initial citation considered. Draw line through citation if not in conformance and no considered. Include copy of this form with next communication to applicant.		

US Serial No. 10/338,452
Clariant GmbH

2002DE402

THREE-PHASE SYSTEMS

[0001] The phase behavior of hydrocarbon/surfactant/water systems is a constituent of numerous investigations. Through the appropriate choice of components, it is possible to obtain three-phase systems which consist of an upper hydrocarbon phase, a middle microemulsion phase of solubilized hydrocarbon, surfactant and water, and a lower water phase. Such three-phase systems are described in

- M. Kahlweit and R. Strey, "The Phase behaviour of H₂O-Oil-Nonionic Amphiphile Ternary Systems" in "Microemulsion Systems", Surfactant Science Series Vol. 24, Dekker, New York, 1987.
- K. Shinoda and S. Friberg, "Emulsions and Solubilization", Wiley, New York, 1986.
- H.G. Hauthal and K. Quitsch, "Neues über Mikroemulsionen" [New findings relating to microemulsions], Z. Chem., 30, 274 - 281 (1990).

[0002] In use, such three-phase systems are limited to technical applications and laboratory uses (tertiary petroleum recovery, media for chemical reactions etc.). A reason for this is the toxicity of the upper hydrocarbon phase (toluene etc.), which does not allow their use in the cosmetics and household sector. If the hydrocarbons are replaced by the oils customary in cosmetics (e.g. mineral oils, polydecenes, triglycerides, natural oils and esters), the average microemulsion phase, and thus the entire three-phase system, becomes thermodynamically unstable.

[0003] Surprisingly, it has now been found that three-phase mixtures which comprise a polyethylene glycol phase instead of the water phase are thermodynamically stable even when the oil phase consists of the oils customary in cosmetics, such as, for example, mineral oils, polydecenes, triglycerides, natural oils and esters. The polyethylene glycol phase may be a polyethylene glycol/water mixture or pure polyethylene glycols.

- [0004] The presence of three phases imparts a very esthetic appearance to the three-phase systems according to the invention. By adding dyes, particularly by adding differently colored water-soluble and oil-soluble dyes, it is possible to emphasize the particular external appearance yet further. The use is not limited to the cosmetics and household sector. Use in the industrial sector, in the laboratory sector, but also as a toy, advertising medium, art object, or else as teaching aid for demonstrating physical phenomena is likewise possible.
- [0005] The invention provides three-phase systems comprising
- a) a polyethylene glycol phase comprising
 - a1) 50 - 100% by weight of at least one polyethylene glycol and
 - a2) 0 - 50% by weight of water,
 - b) an oil phase and
 - c) a microemulsion phase comprising
 - c1) the components of the polyethylene glycol phase a),
 - c2) the components of the oil phase b) and
 - c3) at least one surfactant.
- [0006] Preference is given to three-phase systems which comprise
- a) 10 to 80% by weight, preferably 10 to 50% by weight, of the polyethylene glycol phase,
 - b) 10 to 80% by weight, preferably 10 to 50% by weight, of the oil phase and
 - c) 10 to 80% by weight, preferably 10 to 50% by weight, of the microemulsion phase.
- [0007] Preference is given to polyethylene glycol phases a) which comprise 50 to 100% by weight, preferably 75 to 100% by weight, particularly preferably 85 to 100% by weight, of polyethylene glycol and 0 to 50% by weight, preferably 0 to 25% by weight, particularly preferably 0 to 15% by weight, of water.

- [0008] In a particular embodiment, the polyethylene glycol phase a) comprises 100% by weight of polyethylene glycol.
- [0009] The polyethylene glycol phase a) can comprise one or more polyethylene glycols.
- [00010] The polyethylene glycols preferably have a molecular weight of from 150 to 35 000 g/mol, preferably 200 to 800 g/mol.
- [00011] The surfactants of the microemulsion phase c) may be nonionic, cationic, anionic and/or amphoteric surfactants.
- [00012] The nonionic surfactants are preferably fatty alcohol ethoxylates, dimethylamine oxides, ethoxylated castor oils, alkyl polyglucosides, fatty acid sorbitol esters, fatty acid polyglycerol esters, ethoxylated fatty acid polyglycerol esters, fatty acid monoethanolamide ethoxylates, glycerol mono- and diesters of fatty acids and/or triesters of phosphoric acid.
- [00013] Likewise preferred nonionic surfactants are (C₈-C₂₂)-alkyl or alkenyl ethoxylates having 2 to 20 ethylene oxide groups.
- [00014] The anionic surfactants are preferably monoesters of phosphoric acid, diesters of phosphoric acid, alkyl sulfates, alkyl ether sulfates, preferably sodium laureth sulfate, alkylamidopolyglycol ether sulfates, alkylpolyglycol ether carboxylates, alkylpolyglycol ether sulfosuccinates and/or fatty acid isethionates.
- [00015] The amphoteric surfactants are preferably acyl glutamates, alkylamidopropylbetaines, preferably cocoamidopropylbetaine, fatty acid methyl taurides, fatty acid sarcosides and/or amphotoacetates.

4

[00016] In a particular embodiment, the surfactants are betaines, alkyl ether sulfates or mixtures thereof.

[00017] The three-phase systems preferably comprise 1 to 20% by weight, particularly preferably 5 to 20% by weight, of surfactants.

[00018] Suitable oil phases are preferably mineral oils, polydecenes, triglycerides, e.g. capric/caprylic triglycerides, natural oils, e.g. orange oil and/or esters, preferably stearates, palmitates and myristates.

[00019] Preferred three-phase systems are those in which the components of the oil phase b) are solubilized in the microemulsion phase c) with a degree of solubilization S greater than or equal to 0.8, preferably greater than or equal to 1.5. The degree of solubilization S is the volume ratio of the oil components to the surfactant components.

[00020] In a particular embodiment, the three-phase systems also additionally comprise polar organic compounds, preferably hydroxy compounds and/or polyhydroxy compounds, particularly preferably glycerol, propylene glycol, ethanol, hexylene glycol and/or isopropanol.

[00021] In a particular embodiment, the three-phase systems also comprise water-soluble and/or oil-soluble dyes which are preferably differently colored. This allows advantageous esthetic effects to be achieved.

[00022] The three-phase systems are preferably cosmetic compositions. Particular preference is given to bath oils. Advantageously, the bath oils are shaken prior to use so that they are added to the bath water in the form of an emulsion. Additional ingredients which may be used are the substances customary in cosmetics, such as, for example, perfume oils, ethereal oils, plant extracts, colorants, cationic polymers, solubilization auxiliaries, vitamins and vitamin derivatives, pearlescence-imparting agents,

5

preservatives, skin feel improvers, stabilizers, UV absorbers, hydroxy acids and salts thereof.

[00023] Due to the thermodynamic stability of the three-phase systems according to the invention, they can advantageously be prepared by simply mixing the individual components of phases a), b) and c).

[00024] The thermodynamic equilibrium of the three phases establishes itself. In a preferred embodiment, the hydrophilic components (polyethylene glycols, water, water-soluble dyes etc.), including the surfactants, are mixed together. Separately from this, the hydrophobic, water-insoluble components (oils, oil-soluble dyes etc.) are mixed together. Subsequently, the hydrophilic mixture and the hydrophobic mixture are mixed together.

[00025] Accordingly, the invention also provides three-phase systems obtainable by preparing a mixture comprising

- i) polyethylene glycol(s),
- ii) optionally water,
- iii) oil component(s) and
- iv) surfactant(s).

[00026] Preference is given to three-phase systems obtainable by preparing a mixture comprising

- i) 10 to 70% by weight, preferably 25 to 60% by weight, of polyethylene glycol(s),
- ii) 0 to 70% by weight, preferably 0 to 20% by weight, of water,
- iii) 10 to 70% by weight, preferably 25 to 60% by weight, of oil component(s) and
- iv) 2 to 20% by weight, preferably 5 to 20% by weight, of surfactant(s).

[00027] In a particular embodiment, the three-phase systems are free from water. In a preferred embodiment, the mixtures additionally comprise water-soluble and/or oil-soluble dyes.

6

[00028] The preferred polyethylene glycols, oil components and surfactants are the compounds already described above.

Examples

[00029] In the examples, the degree of ethoxylation of the surfactants was optimized such that approximately equal amounts of oil phase and hydrophilic phase were solubilized in the microemulsion. S is the degree of solubilization of the oil on the basis of the volume ratio. Oleth-8 and Oleth-10 are the INCI names for oleyl alcohol polyglycol ether having 8 or 10 mol, respectively, of ethylene oxide. PEG-8 is the INCI name for polyethylene glycol having an average molar mass of 400 g/mol.

[00030] The quantitative data is in % by weight.

7

Examples 1 to 3:

Ex.	Polyethylene glycol phase		Surfactant		Oil phase	Phase volume			S
	PEG-8	Water	Oleth-8	Oleth-10	Mineral oil	Bottom	Middle	Top	
1	43.1	10.8	6.9	0	39.2	28.9	34.8	36.3	1.5
2	27.2	6.8	17.3	0	48.7	14.0	82.0	4.0	2.2
3	36.9	15.8	9.0	0.2	38.1	15.5	62.5	22.0	2.5

Example 4: Three-phase bath oil

	Component	% by wt.
Hydrophilic components	PEG-8	41.8
	Water	10.5
Surfactants	Oleth-5 ([®] Emulsogen LP)	4.4
	Coceth-10 ([®] Genapol C-100)	5.3
Hydrophobic components	Mineral oil	29.8
	Capric/caprylic triglycerides	7.2
	Orange oil	0.4
	Vitamin E acetate	0.4
Preservative	Nipaguard PDU	0.3
Water-soluble dye	Vitasyn Ponceau 4R82	q.s.
Oil-soluble dye	Beta-carotene	q.s.

[00031] The bath oil separated into the following three phases:

upper phase (red) 28.5% by volume
middle phase (orange) 48.0% by volume
lower phase (yellow) 23.5% by volume

[00032] The preparation of examples 1 to 4 was carried out by preparing a mixture of the hydrophilic components and, separately, a mixture of the

8

hydrophobic components. Subsequently, the surfactants were added to the hydrophilic mixture. Finally, the hydrophilic mixture and the hydrophobic mixture were mixed together.

2002DE402

9

Claims:

1. A three-phase system comprising
 - a) a polyethylene glycol phase comprising
 - a1) 50 - 100% by weight of at least one polyethylene glycol and
 - a2) 0 - 50% by weight of water,
 - b) an oil phase and
 - c) a microemulsion phase comprising
 - c1) the components of the polyethylene glycol phase a),
 - c2) the components of the oil phase b) and
 - c3) at least one surfactant.
2. The three-phase system as claimed in claim 1, which comprises
 - a) 10 to 80% by weight, preferably 10 to 50% by weight, of the polyethylene glycol phase,
 - b) 10 to 80% by weight, preferably 10 to 50% by weight, of the oil phase and
 - c) 10 to 80% by weight, preferably 10 to 50% by weight, of the microemulsion phase.
3. The three-phase system as claimed in claim 1 and/or 2, wherein the polyethylene glycol phase a) comprises 50 to 100% by weight, preferably 75 to 100% by weight, particularly preferably 85 to 100% by weight, of polyethylene glycol and 0 to 50% by weight, preferably 0 to 25% by weight, particularly preferably 0 to 15% by weight, of water.
4. The three-phase system as claimed in claim 3, wherein the polyethylene glycol phase a) comprises 100% by weight of polyethylene glycol.
5. The three-phase system as claimed in at least one of claims 1 to 4, wherein the polyethylene glycols of the polyethylene glycol phase a) have a molecular weight of from 150 to 35 000 g/mol, preferably 200 to 800 g/mol.

6. The three-phase system as claimed in at least one of claims 1 to 5, wherein the surfactants of the microemulsion phase c) are nonionic, cationic, anionic and/or amphoteric surfactants.

7. The three-phase system as claimed in claim 6, wherein the nonionic surfactants are fatty alcohol ethoxylates, dimethylamine oxides, ethoxylated castor oils, alkyl polyglucosides, fatty acid sorbitol esters, fatty acid polyglycerol esters, ethoxylated fatty acid polyglycerol esters, fatty acid monoethanolamide ethoxylates, glycerol mono- and diesters of fatty acids and/or triesters of phosphoric acid.

8. The three-phase system as claimed in claim 6, wherein the nonionic surfactants are (C₈-C₂₂)-alkyl or alkenyl ethoxylates having 2 to 20 ethylene oxide groups.

9. The three-phase system as claimed in claim 6, wherein the anionic surfactants are monoesters of phosphoric acid, diesters of phosphoric acid, alkyl sulfates, alkyl ether sulfates, preferably sodium laureth sulfate, alkylamidopolyglycol ether sulfates, alkylpolyglycol ether carboxylates, alkylpolyglycol ether sulfosuccinates and/or fatty acid isethionates.

10. The three-phase system as claimed in claim 6, wherein the amphoteric surfactants are acyl glutamates, alkylamidopropylbetaines, preferably cocoamidopropylbetaine, fatty acid methyl taurides, fatty acid sarcosides and/or amphotoacetates.

11. The three-phase system as claimed in claim 6, wherein the surfactants are betaines, alkyl ether sulfates or mixtures thereof.

12. The three-phase system as claimed in at least one of claims 1 to 11, wherein the components of the oil phase b) are mineral oils, polydecones,

11

triglycerides, e.g. capric/caprylic triglycerides, natural oils, e.g. orange oil and/or esters, preferably stearates, palmitates and myristates.

13. The three-phase system as claimed in at least one of claims 1 to 12, wherein the components of the oil phase b) are solubilized in the microemulsion phase c) with a degree of solubilization S greater than or equal to 0.8, preferably greater than or equal to 1.5.

14. The three-phase system as claimed in at least one of claims 1 to 13, which comprises polar organic compounds, preferably hydroxy compounds and/or polyhydroxy compounds, particularly preferably glycerol, propylene glycol, ethanol, hexylene glycol and/or isopropanol.

15. The three-phase system as claimed in at least one of claims 1 to 14, which comprises at least one water-soluble dye.

16. The three-phase system as claimed in at least one of claims 1 to 14, which comprises at least one oil-soluble dye.

17. The three-phase system as claimed in at least one of claims 1 to 14, which comprises at least one water-soluble dye and at least one oil-soluble dye.

18. The three-phase system as claimed in claim 17, wherein the water-soluble dyes and the oil-soluble dyes are differently colored.

19. The three-phase system as claimed in at least one of claims 1 to 18, in the form of a cosmetic composition.

20. The three-phase system as claimed in claim 19, in the form of a bath oil.

21. The three-phase system obtained by preparing a mixture comprising

12

- i) polyethylene glycol(s)
- ii) optionally water
- iii) oil component(s) and
- iv) surfactant(s).

22. The three-phase system as claimed in claim 21, obtainable by preparing a mixture comprising

- i) 10 to 70% by weight, preferably 25 to 60% by weight, of polyethylene glycol(s),
- ii) 0 to 70% by weight, preferably 0 to 20% by weight, of water,
- iii) 10 to 70% by weight, preferably 25 to 60% by weight, of oil component(s) and
- iv) 2 to 20% by weight, preferably 5 to 20% by weight, of surfactant(s).

23. The three-phase system as claimed in claim 21 and/or 22, wherein the polyethylene glycols i) have a molecular weight of from 150 to 35 000 g/mol, preferably 200 to 800 g/mol.

24. The three-phase system as claimed in at least one of claims 21 to 23, wherein the oil component iii) is mineral oils, polydecenes, triglycerides, e.g. capric/caprylic triglycerides, natural oils, e.g. orange oil and/or esters, preferably stearates, palmitates and myristates.

25. The three-phase system as claimed in at least one of claims 21 to 24, which additionally comprises water-soluble and/or oil-soluble dyes which are preferably differently colored.

26. The three-phase system as claimed in at least one of claims 21 to 25, in the form of a cosmetic composition, preferably a bath oil.

2002DE402

13

Abstract**Three-phase systems**

The invention relates to three-phase systems comprising

- a) a polyethylene glycol phase comprising
 - a1) 50 - 100% by weight of at least one polyethylene glycol and
 - a2) 0 - 50% by weight of water,
- b) an oil phase and
- c) a microemulsion phase comprising
 - c1) the components of the polyethylene glycol phase a),
 - c2) the components of the oil phase b) and
 - c3) at least one surfactant.

The three-phase systems are thermodynamically stable and are preferably suitable as cosmetic compositions, particularly preferably as bath oils.

MP



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Dr. Kern
Dr. Hennig
6.1.25.11.03 OK

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Anmeldung Nr./Application No./Demande n°/Patent Nr./Patent No./Brevet n°.

03022145.1-2113-

Anmelder/Applicant/Demandeur/Patentinhaber/Proprietor/Titulaire

Clariant GmbH

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Das Europäische Patentamt übermittelt beiliegend den europäischen Recherchenbericht zu der obengenannten europäischen Patentanmeldung.

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☒ Zusammenfassung

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Die folgende Abbildung wird mit der Zusammenfassung veröffentlicht: KEINE

T-ART	FRIST
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Falls Artikel 10 der Gebührenordnung in Anwendung kommt, ergeht noch eine gesonderte Mitteilung der Eingangsstelle hinsichtlich der Rückerstattung der Recherchegebühr.



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EUROPÄISCHER TEILRECHERCHENBERICHT

der nach Regel 45 des Europäischen Patent-
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Nummer der Anmeldung

EP 03 02 2145

EINSCHLÄGIGE DOKUMENTE			
Kategorie	Kennzeichnung des Dokuments mit Angabe, soweit erforderlich der maßgeblichen Teile	Betrifft Anspruch	KLASSIFIKATION DER ANMELDUNG (Int.Cl.7)
P, X	EP 1 327 439 A (CLARIANT GMBH) 16. Juli 2003 (2003-07-16) * Absätze '0005!-'0019!', '0022!-'0030! *	1-14	B01F17/42 B01F17/00 A61K7/46
A	DE 196 12 085 A (HENKEL KGAA) 2. Oktober 1997 (1997-10-02) * Ansprüche *	1, 6, 13, 14	
A	DE 38 23 539 A (MIFA AG FRENKENDORF) 26. Januar 1989 (1989-01-26) * Anspruch 1 *	1, 6, 13, 14	
A	US 2001/036450 A1 (SIMONNET JEAN-THIERRY ET AL) 1. November 2001 (2001-11-01) * Ansprüche *	1, 6, 13, 14	
			RECHERCHIERTE SACHGEBIETE (Int.Cl.7)
			B01F A61K
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1-14

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Die Recherche wurde aus folgenden Gründen auf die Beispiele 1 und 2 begrenzt:

In Anspruch 1 fehlt bei der Formel $R-(OCHR'CH)nOH$ die Definition des vierten Substituenten an der $-CH-$ Gruppe. Da es nicht unmissverständlich klar ist, ob dieser vierte Substituent Wasserstoff, also $R-(OCHR'CH_2)nOH$, oder R' , also $R-(OCHR'CHR')nOH$ sein soll und die gleiche Unklarheit auch in der Beschreibung besteht, scheint dieser Fehler irreparabel zu sein. Lediglich die INCI-Bezeichnungen "Oleth-5, -8 und -10" sind hinreichend klar und können recherchiert werden.

Ein weiteres, gravierendes Problem in der Anmeldung besteht darin, dass Anspruch 6, der als von Anspruch 1 abhängig formuliert wurde, in Wahrheit den Umfang von Anspruch 1 durch die Erweiterung der Komponente d) aus Anspruch 1 auf jede denkbare Art von Tensiden soweit ausdehnt, dass er, Anspruch 6, den breitesten Anspruch darstellt. Mithin fällt nun jede Zusammensetzung unter den Gegenstand der Anmeldung, die eine wasserunlösliche Flüssigkeit, PEG und irgendein Tensid enthält. Die Recherche ergab daher eine sehr große Zahl neuheitsschädlicher Dokumente. Diese Zahl ist so groß, daß sich unmöglich feststellen lässt, für was in der Gesamtheit der Patentansprüche eventuell nach zu Recht Schutz begehrt werden könnte (Art. 84 EPÜ). Aus diesen Gründen erscheint eine sinnvolle Recherche über den gesamten Bereich der Patentansprüche unmöglich.

ANHANG ZUM EUROPÄISCHEN RECHERCHENBERICHT ÜBER DIE EUROPÄISCHE PATENTANMELDUNG NR.

EP 03 02 2145

In diesem Anhang sind die Mitglieder der Patentfamilien der im obengenannten europäischen Recherchenbericht angeführten Patentedokumente angegeben.
Die Angaben über die Familienmitglieder entsprechen dem Stand der Datei des Europäischen Patentamts am
Diese Angaben dienen nur zur Unterrichtung und erfolgen ohne Gewähr.

11-11-2003

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Für nähere Einzelheiten zu diesem Anhang : siehe Amtsblatt des Europäischen Patentamts, Nr.12/82

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